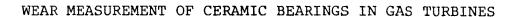


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A.J. Armini, Ph.D. S.N. Bunker, Ph.D.

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IMPLANT SCIENCES CORPORATION
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MARCH 1990

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The primary findings of the program are:

- a. The method for tagging Si_3N_4 , SiC, and M50 bearing components to depths of interest in bearings (1-20 microns) was developed.
- b. Calculated techniques were developed to predict activation and count rates in the detector system for both the pin-on-disk and bearing test rig.
 - c. The data acquisition electronics were selected.
- d. Radiation safety issues were examined and found to be not a problem since activity levels required were below values exempt from regulation by the NRC or the States.
- e. The use of the technique was deamed feasible and useful for measuring needed wear parameters in materials under conditions important to ceramic bearing operation.

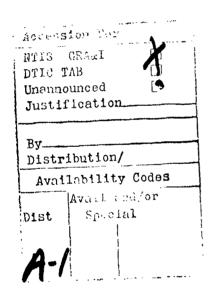




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SECTION 1 INTRODUCTION

Ceramic bearing technology is rapidly becoming a viable option in the design of high temperature gas turbines. However, much of the basic design data on the tribiological properties of these materials is still lacking.

The objective of this program was to determine the feasibility of measuring some of the wear properties of ceramic bearing materials in real time without disturbing the wear couple or the operating conditions, such as temperature or break-in geometry.

The method chosen is to selectively introduce a radioactive tag into the surface of a ceramic part and to measure the wear amount by monitoring the strength of the tagging activity as the test progresses. Although this method, called Surface Layer Activation (SLA), has been used for many years in the automobile (1)(2) and heavy machinery industries (3)(4), it has not yet been used to measure the extremely minute amounts of wear which are experienced by ball bearings.

This program was analytical in nature, and its principal task was to show the feasibility and accuracy of such a technique applied to Si₃N₄ and SiC ceramic bearing components. In addition the goal was to develop the analytical theory, computer software and operational techniques needed for wear tests.

The primary findings of the program are:

- l. The method for tagging $\mathrm{Si_3N_4}$, SiC , and M50 bearing components to depths of interest in bearings (1-20 microns) was developed, and subcontractors with the capability to implant these isotopes were identified.
 - 2. Calculational techniques were developed to predict

activation and count rates in the detector system for both the pinon-disk and bearing test rig.

- 3. The data acquisition electronics were selected and vendors identified.
- 4. Radiation Safety issues were examined and found to be not a problem. Activities required were below values exempt from regulation by the NRC or the States.
- 5. The use of the technique was proven feasible and valuable in measuring needed wear parameters in materials and conditions important to ceramic bearing operation.

SECTION 2 BACKGROUND

The ball and roller bearings used in gas turbines are generally made of steels (M50, 52100), and loss of bearing performance due to mild wear is not a normal failure mode. liquid lubricated bearings, the lubricant serves to provide full separation of the rolling element and raceway which prevents wear. The lubricant also acts as a convective heat transfer medium to carry the heat out of the bearing and to equalize the temperature of the various components. Ceramic bearings, either non-lubricated or with solid lubricants, are unlikely to ever operate at full separation between the contact surfaces, and therefore mild wear is of concern. For example, Bunting (5) has shown that a change in raceway conformity of an all ceramic bearing from 0.54 (as machined) to 0.505 due to mild wear can lead to thermal run away Thus the measurement of mild wear in a and bearing seizure. ceramic bearing is much more important and probably should be monitored in real time to prevent catastrophic failure in flight service.

In addition to this special vulnerability of ceramic bearings to mild wear, the tribiological properties are not well understood and the amount of test data available is sparse compared to what is presently known about bearing steels. Clearly there is a need to accumulate basic tribological and bearing test rig data on the performance of ceramic bearing materials under various startup loads and lubrication schemes as well as various operating temperatures.

The SLA activation technique is capable of providing wear rate data in real time from initial break-in to final equilibrium without disassembling the wear couple. The proposed thin layer activation method was developed several years ago to make in-situ

measurements of the surface wear of parts in mechanical systems. It is particularly suited to the measurements of surface wear in areas which are generally inaccessible when real-time data are required, or where conditions make other physical measurement methods impossible. The original application of the technique, invented by the principle investigator while at McDonnell Douglas Corporation in 1971, was to measure the erosion of missile nosetips during atmospheric re-entry. The system required wear measurements to be made during the flight itself since no worn part could be recovered. The basic structure of the measurement system, as shown in Figure 2-1, consisted of two parts: an activated volume of the test component and a Sodium Iodide gamma-ray detector. The nosetip was first made radioactive by the bombardment of a proton beam which produced Be from the carbon material. In flight, the detector measured the radiation being emitted by the activated material, which was a function of the thickness of material remaining. This radiation measurement, or count rate, was related to the exact eroded depth by a calibration curve obtained prior to the missile flight.

In applications involving mechanical systems, the concept is similar. The wearpoint of interest is either directly bombarded with a high energy beam of nuclei from a suitable atom accelerator, or radioactive atoms are embedded in the test sample as a secondary scattering process. The geometries involved will be shown in a later section, but in either case, the result is a shallow layer of lightly doped radioactive tracer atoms that can serve as an indicator of thickness removed. Figure 2-2 shows how the technique has been applied to real-time measurement of wear in a piston ring. Figure 2-3 shows how the gamma rays are detected outside of the automobile engine while it is running.

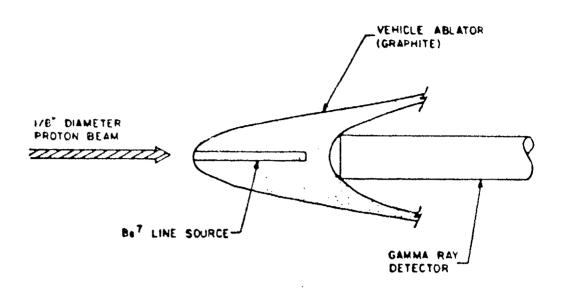


FIGURE 2-1. ORIGINAL APPLICATION OF THE SLA TECHNIQUE TO NOSETIP EROSION DURING RE-ENTRY.

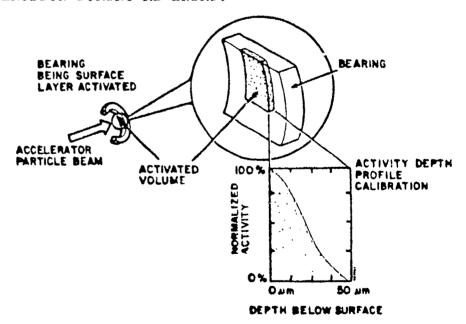


FIGURE 2.2. PISTON RING ACTIVATION OF A THIN SURFACE LAYER SHOWING THE ACTIVATION DEPTH PROFILE.

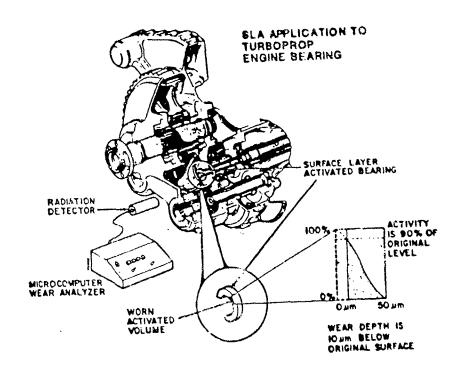


FIGURE 2-3. OUTSIDE RACE IN OPERATING POSITION BEING CONTINUOUSLY MONITORED BY A GAMMA RAY DETECTOR OUTSIDE OF A TURBOPROP ENGINE.

SECTION 3 SELECTION OF BEARING WEAR EXPERIMENTS

The experimental program will attempt to simulate wear on an all ceramic $\mathrm{Si}_3\mathrm{N}_4$ or SiC bearing as well as wear of the metal races in a hybrid bearing. In addition, two type of wear on the ceramic will be investigated. Wedeven (6) at SKF has shown two distinct types of wear debris generated by $\mathrm{Si}_3\mathrm{N}_4$ rolling contact. At high contact stress with traction, Hertzian stress cracks form at the edges of the contact area and cause fracture. These conditions cause large particle spallation resulting in larger size particles ranging from 2 microns to 5 microns.

At lower loads, the surface becomes highly burnished and thin films of transparent wear debris of extremely small size are generated. This fine debris has been shown $^{(7)}$ to be amorphous hydrated silicon oxide (SiO/XH₂O), which is softer than Si₃N₄ and may be a significant solid lubricant in high temperature dry operation.

The high contact stress wear can be measured using a pin-on-disk wear apparatus where the elements in the ceramic can be activated using the reaction:

$$N^{14} + P \longrightarrow Be^7 + 2He^4$$

in Silicon Nitride or

$$C^{12} + P \longrightarrow Be^7 + p+n+He^4$$

in Silicon Carbide

This will produce a surface layer about 10 to 50-microns deep which is doped with Be 7 , a gamma emitter with a 53 day half-life. At lower contact stresses a second type of activation can be used to produce an ultra-shallow, 1-micron (40-microinch) layer containing radioactive Be 7 . This type of activation can be used to detect the presence of the burnishing wear, which produces very fine SiO $_x$ sheets about a few millionths of an inch thick.

Table 3-1 shows a summary of the types of test apparatus that might be used for the activation methods. The pin-on-disk apparatus is already in existence at Implant Sciences Corporation. The high load bearing test could be conducted using Government test facilities.

TABLE 3-1. PLANNED WEAR TEST MATRIX FOR PHASE II PROGRAM

| TEST APPARATUS | LIGHT LOAD | HEAVY LOAD |
|----------------------------------|-------------------------|---|
| pin-on-dick | Be ⁷ implant | Be ⁷ in Ceramic disk Direct Co ⁵⁶ in M50 |
| High load Bearing Test rig | N, A | Be ⁷ in ceramic race Direct Co ⁵⁶ in M50 |

3.1 PIN-ON-DISK TEST

The pin-on-disk wear tester at Implant Sciences Corporation is shown in Figure 3-1. This tester has a contact force variable up to 10 GPa and can also measure sliding friction coefficients at velocities up to 40 cm/s.

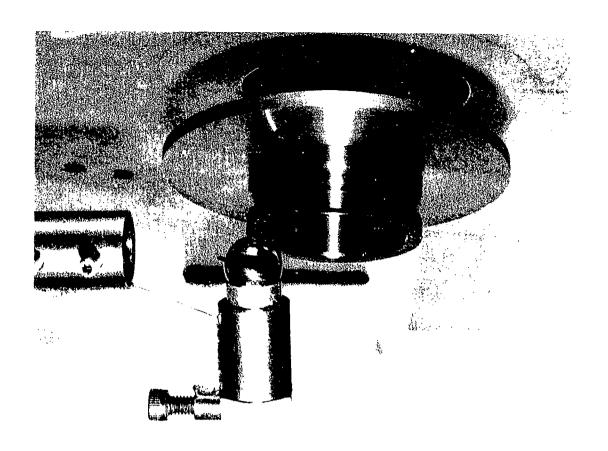


FIGURE 3-1. PHOTOGRAPH OF IMPLANT SCIENCES' PIN-ON-DISK TESTED WITH CERAMIC BALL ON M50 DISK.

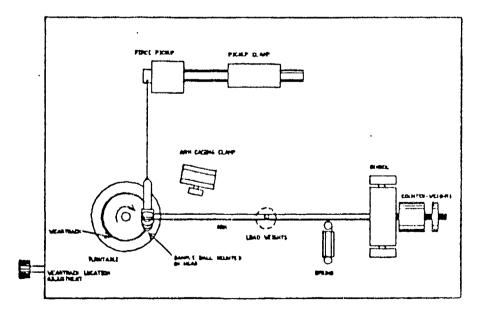


FIGURE 3-2. DIAGRAM OF IMPLANT SCIENCES' PIN-ON-DISK TESTER (TOP VIEW)

The tester can also be operated in a transparent glove box so that testing can be done in atmospheres other than air. Thermal insulation and a "clam shell" heater can also be added, so that the pin and disk can be raised to a temperature of up to 600°C. Figure 3-2 shows a plan-view diagram of the tester.

The pin-on-disk tests can be designed to simulate operational loads and sliding speeds. To this end we have selected the same silicon nitride bearing size and conditions described by Bunting (5) of SKF for our feasibility analysis. This paper describes a test of an all ceramic bearing with the following parameters:

| OD of inner race | 36 mm |
|--------------------------|-------------------|
| Speed | 38,000 r/min |
| Hertzian stress | 2.07 GPa |
| Temperature | up to 600°C |
| Test duration | up to 246 minutes |
| Calculated sliding speed | 1.32 m/s (1.8%) |
| Total race way wear | 13 µm (10 balls) |
| | |

Since a pin-on-disk test is 100% sliding rather than 1.8%, the equivalent sliding distance turns out to be 1.95 x 10⁴ meters which could be simulated in 28.7 hours at 100 r/min. Thus on Implant Science Corporation's pin-on-disk apparatus for Silicon Nitride using 2.07 GPa Hertzian stress and 100 r/min disk speed, a 28.7 h. run should yield about 1 micron of wear on the disk (at 600°C). This simulation and predicted wear depth will be used in the next section to determine the nuclear tagging method to be used.

Table 3-2 shows the total scope of all test conditions available with the present pin-on-disk apparatus. If one uses three values for each parameter, then the total number of tests could be 3^5 or 243. A more realistic test matrix has 12 tests and is shown in Table 3-3. These test conditions, a subset of Table

3-2, were selected to test the maximum capabilities of each of the three wear couples chosen. The final set of test conditions will be selected with consultation and approval of the AF project technical officer.

For each of these tests the disk wear as a function of time can be measured using the nuclear wear gauge. This can be done without stopping the rotation and therefore distributing the break-

TABLE 3-2. SCOPE OF PIN-ON-DISK TEST CONDITIONS

| <u>Parameters</u> | Test Conditions |
|-------------------|--|
| Hertz stress | 0.2 → 2 GPa |
| Speed | 10 |
| Temperature | $20^{\circ}C \longrightarrow 600^{\circ}C$ |
| Lubrication | none> powder |
| Wear couple (1) | ceramic pin/ceramic/disk |
| (2) | ceramic pin/M50 disk |
| (3) | M50 pin/ ceramic disk |

TABLE 3-3. SELECTED TEST CONDITIONS

| | Hertz Stress | Speed | Temp | | iskwear kpected |
|--------------------------|-----------------|-------|--------------|----------------|--------------------|
| ceramic pin/ceramic disk | 2 GPa | 100 | 650° 20° | none powde: | l µm |
| ceramic pin/M50 disk | 2 | 100 | 300°C 20° | none powder | 10 איים |
| M50 pin/ceramic disk | 2 | 100 | 300°C 20° | none powder | l µm |

in conditions. Data can be taken typically every hour except that during the initial start-up and break-in period data may be taken every 1 minute in order to determine break-in wear rates. Wear cannot be measured on the pins. In a pin-on-disk test, unlike a ball bearing, the same point on the pin is always in contact, a condition which can generate untypical wear rates and surface temperatures. The disk, on the other hand has a ring wear groove which has time to recover and cool during each revolution.

In addition to the nuclear wear measurement, the friction force and the temperature will be measured during each test.

3.1.1 PIN-ON-DISK TEST GEOMETRY

The Implant Science Corporation's pin-on-disk tester ordinarily uses a ball, typically 1/2 ins diameter as the pin and the flat side of a bearing ring as the disk. Because of the SLA technique, the ball geometry must be changed to a cylinder or a roller. The reason for this is illustrated in Figure 3-3.

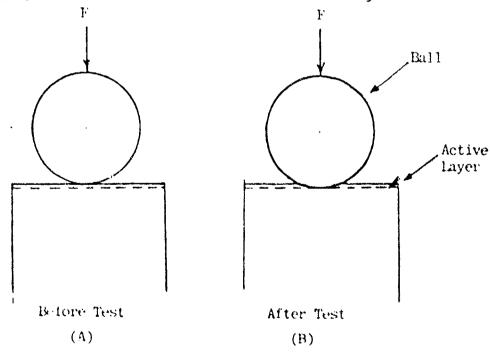


FIGURE 3-3. BALL BEARING ON FLAT DISK. LEFT SIDE BEFORE TEST, RIGHT SIDE AFTER WEAR GROOVE WORN.

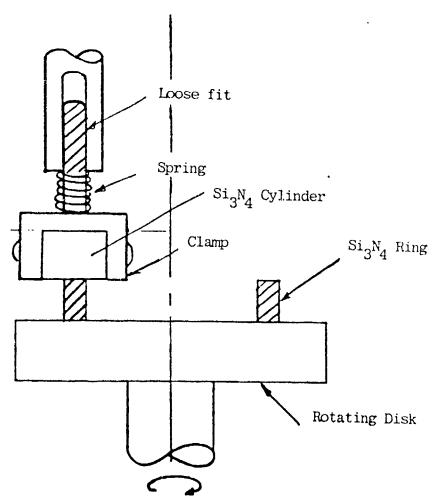


FIGURE 3-4. SCHEMATIC OF STATIONARY ROLLER (PIN) ON SLA ACTIVATED RING (DISK).

Before the test the activated surface is of uniform thickness, parallel to the ring edge. When the ball wears the disk (the ball wears too), the activity is not removed in level manner. The calibration procedure to be described in Section 6 requires the wear front to be perfectly parallel to the P surface. In order to wear the top of the ring and the pin in the same manner as the calibration procedure, it is necessary to use the side of a roller as the pin. This insures that the ring will not wear only in a narrow line where the ball touches it. Since it is critical that the roller is always seated parallel to the ring (the roller doesn't roll in this test, it only slides) it must be able to tilt from side to side to always rest parallel to the top surface of the ring. Figure 3-4 shows the method which can be used to achieve this.

The apparatus will also have a pair of clam-shell resistance heaters around the disk to heat the disk and roller up to 600°C. The gamma ray detector can view the ring from the side through the two Aluminum reflectors and thermal insulators. There should also be a vacuum pickup continuously sucking the wear debris away from the ring. The analysis of the count rates and accuracies of the measurement will be discussed in Section 4.

3.2 HIGH SPEED BEARING RIG TESTS

For this analysis we have tentatively selected an all cerumic roller bearing of approximately 30 mm bore and 62 mm O.D. for ease of activation. The inside race would be activated in a 1/8 in wide strip in the center of the O.D.

The narrow band of activity insures that the roller will wear the activity off uniformly, which is also the reason for selecting a roller rather than a ball bearing. At high speeds, a roller bearing operates close to a mode known as outer raceway control. This is caused by greater ball loading on the outer raceway due to centrifugal force, and it makes most of the slip or spin occur on the inner raceway, resulting in local heat and wear. For this reason we have selected the inner raceway as the nuclear tagged surface.

Following the analytical method of Bunting (5) we project that about 13 to 15 microns of wear can occur before heat generation becomes intolerable. On this same size bearing, Bunting computes that the as-machined conformity of 0.54 becomes 0.505 with 13 micron of wear from the inner race. Figure 3-5 shows that thermal runaway is starting to occur at a conformity of 0.51.

In order to activate Silicon Nitride or Silicon Carbide to a depth of 2 to 13 microns, the receil implant technique will be used. This method, which will be described in more detail later,

uses an auxillary nuclear reaction to generate high energy Be⁷, which is then implanted into the target.

The reaction is:

$$Li^7 + B^1 \longrightarrow Be^7 + n$$

After the inner race is implanted, the actual test configuration will be similar to the bearing test rig shown in Figure 3-6.

Although this is not a schematic of an available test rig, it illustrates the shielding and detector distance involved in such a test. The gamma ray detector parameters for this candidate test are:

Detector distance= 8 in Steel thickness=1/2 in Detector Diameter= 6 in Si_3N_4 thickness=3/4 in Detector Eff= 0.8

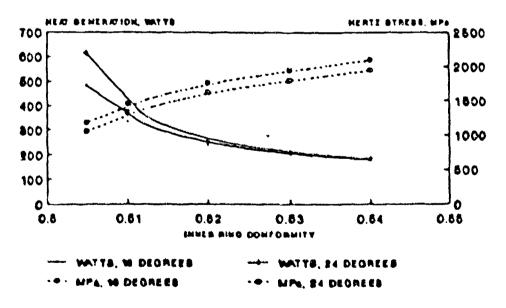


FIGURE 3-5. INNER RING HEAT GENERATION AND HERTZ STRESS AS A PUNCTION OF INNER RING CONFORMITY AND CONTACT ANGLE.

Using the computer program developed during this program, (see Appendix A) the total inner race activity for a count rate of 100 cps is 0.6 microcurie. This is a small amount of activity and should pose no great problem in performing such a test. It is in fact below the limit of regulation by the NRC. (See Section 8)

The detailed method of activation and calibration of this inner race will be covered in section 4 and 6.

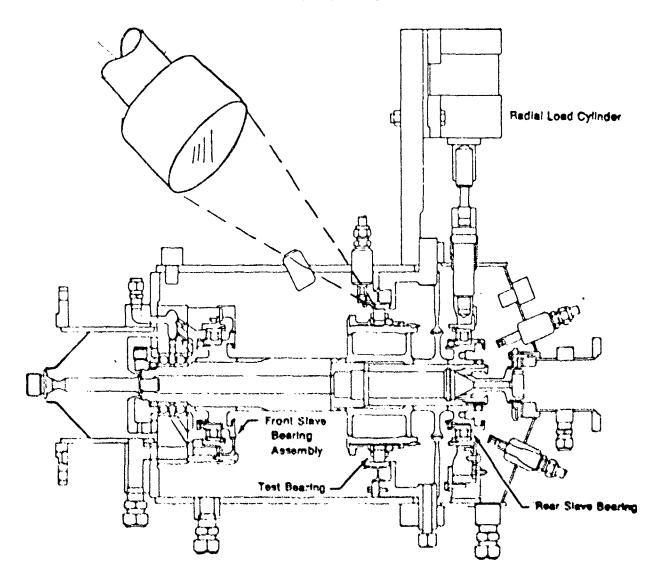


FIGURE 3-6. SCHEMATIC OF TYPICAL BEARING TEST RIG AND GAMMA RAY DETECTOR IN POSITION.

SECTION 4

DETERMINATION OF NUCLEAR TAGGING METHODS AND DETECTION

Measurements of surface material loss have been performed using a variety of nuclear activation techniques which differ mainly in the volume of sample material which is made radioactive.

The SLA technique differs from the older neutron activation method in that only the near surface zone of a part is activated. SLA activation is accomplished by using a directed beam of ions, which have only a very shallow depth of penetration into the part. Neutron activation is by total immersion in which the entire mass of the part is activated. The additional activated volume, essentially all of the sample, is not necessary for the measurement and only serves to increase background and impose serious safety problems.

A typical 3 in OD race will have a layer 10 microns thick by 1/8 in wide when activated using the SLA technique. The radioactivity in this volume is volume 0.5 microcurie and is quite satisfactory for an SLA measurement. An equivalent activation density and measurement sensitivity using thermal neutrons would require the total source strength to be approximately 2500 times greater, and this is a significant strength source.

To achieve these extremely shallow implants of active material two methods are used:

- (1) Direct nuclear reaction in the part (useful from 1 to 1000 micron depths)
- (2) Implantation of active recoil atoms from an auxiliary reaction (useful 0.3 to 10 micron depths)

4.1 DIRECT NUCLEAR REACTION METHOD

In this method a nuclear reaction with a constituent atom in

the part is used to manufacture an active atom. For example, in steels, the iron atoms are reacted with an incoming beam of protons and some of them (a very small fraction) are transmuted to ${\rm Co}^{56}$, which is unstable and emits gamma rays. The reaction can be written:

$$p + Fe^{56} \longrightarrow co^{56} + n$$

This requires a minimum of 10MeV protons for the reaction to occur. This reaction has been used by many workers to measure wear in steels. Similarly, Silicon Nitride or Silicon Carbide can be reacted with protons to form Be⁷ or Na²² as desired. Part A in Table 4-1 shows the reactions of interest in this program for activating ceramic or steel bearing materials.

Table 4-1. Nuclear Tagging Reactions

| (A)Direct Methods | Typical Depth Micron | Half- Life | Gamma Ray Energy | Activation Rate |
|---|----------------------------|---------------|------------------------|--------------------|
| C ¹² (p,pnRe ⁴)Be ⁷ | 10-500 | 53 đ | 0.5MeV | med |
| R ¹⁴ (p,2He ⁴)Be ⁷ | 10-500 | 53đ | 0.5MeV | iow |
| $\operatorname{Si}^{28}(p,\operatorname{Be}^7)\operatorname{Na}^{22}$ | 5-100 | 2.7yr | 1.27MeV | low |
| Fe ⁵⁶ (₁ ,n)Co ⁵⁶ | 10-30 | 77d | lMeV | high |
| (B) <u>Fecoil Methods</u> | | | | |
| $H^1(Li^7, Be^7)n$ | 1-10 | 5 3 đ | 0.5MeV | high |
| $c^{12}(\kappa^{14}, Na^{22}) He^4$ | 0.5-5 | 2.7yr | 1.27MeV | med |
| Fe ⁵⁶ (1,Co ⁵⁶)n | 0.3-1 | 7 7đ | lMeV | low |

For the pin-on-disk tests described in Section 3, a direct activation of the Nitrogen in Silicon Nitride was proposed to form ${\rm Be}^7$ to a depth of 10 microns.

Figure 4-1 shows the geometry used for the calculation of count rate in the pin-on-disk test. The activity counted by the detector A, is given by

$$A = A_{\circ}$$
 $= e^{-\mu pt}$

where:

Ao is the Be⁷ activity induced in the disk

() is the solid angle to the detection

(a) Jobb is the gamma ray attenuation through the shielding

(is the detector efficiency for Be⁷ gamma rays

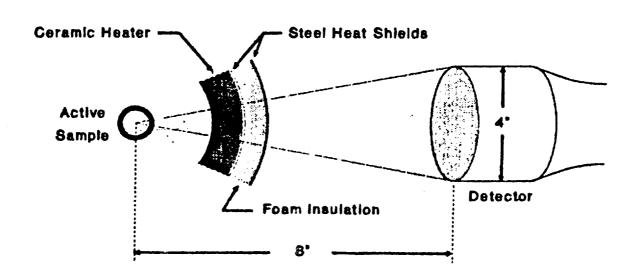


FIGURE 4-1. GEOMETRY FOR CALCULATION OF COUNT RATE IN PIN-ON-DISK

The details of this calculation and a computer program written to solve this equation can be found in Appendix A.

The results show that a Be⁷ actuative of only 0.5 microcuries can give a count rate of 300/s. This means that if a measurement is made for 1 minute per sample, there will be a statistical accuracy of 0.7%. The overall accuracy is also dependent upon the accuracy of the calibration curve, which will be discussed in Section 6.

4.2 THE RECOIL IMPLANTATION METHOD

In this method the nuclear reaction induced by the proton beam occurs in an auxiliary target, and the part in question "catches" the reaction products by being placed close to the target. The geometry is shown below in Figure 4-2.

This method was pioneered by T.W. Conlon at Harwell Laboratory (England). This method is particularly useful when the wear depth is very shallow (1-10 microns) or when the part cannot take the direct heat of the proton beam, such as for plastics or glass.

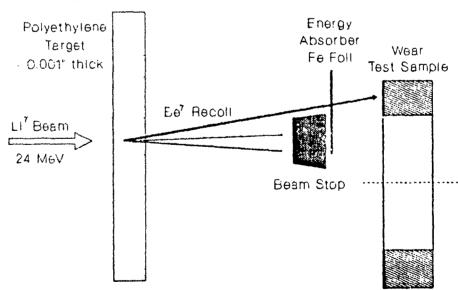


FIGURE 4-2 GEOMETRY USED DURING ACTIVATION BY RECOIL IMPLANTATION OF BE⁷

The recoil reactions of interest for this program are shown in Table 4-1 part B. For the full-scale Silicon Nitride bearing tests described in Section 3.2, the reaction

$$H^1(Li^7, Be^7)$$
 n

has been chosen for quantitative analysis. In this case a polyethylene target is bombarded by a Li⁷ beam from a tandem Van de Graaff accelerator. Using this reaction with 24 MeV Li⁷ incident, the energy spectrum of Be⁷ atoms impinging on the wear part prior to any energy attenuating foils is shown in Figure 4-3. The spectrum is input into Implant Sciences PROFILE CODE, and the calculated implanted depth profile of Be⁷ atoms is shown in Figure 4-4. An energy absorbing foil convenient adjusts the Be⁷ from the surface to a depth of 10 microns for a ceramic bearing test. The calibration curve would then have a resolution of about 0.05 micron per point. This resolution should be sufficient to measure wear rates during break-in as well as during equilibrium.

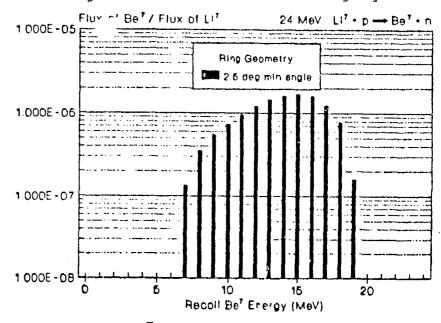


FIGURE 4-3. SPECTRUM OF BE⁷ NUCLEAR GENERATED BY INELASTIC RECOIL REACTION.

| TARGET= | 158 7.87G | 5° keV | ion | Dose/cn2 | keV | Ion | Dose/cm2_ |
|-----------------|-------------------|--------------|-------|--------------------------|---|---------|----------------------|
| Calc. Type Mul | ltilayer Gaussian | 1 7500 | Be- 7 | 4.85e9 | 6 13888 | Be- 7 | 1.46e18 |
| | 888. 1.44c14 .888 | | Be- 7 | 5.46e9 | 7 14888 | | 1.68e18 |
| Sput. Loss Coat | t 8å, Sub 8 | å 3 10800 | Be- 7 | 7.38e9 | 8 15888 | Be~ 7 | 1.78e18 |
| Retn. Dose 1.2 | 24e11/cm2 188.8% | 4 11888 | Be- 7 | 9.59e9 | 9 16080 | Be- 7 | 1.68e18 |
| | | 5 12000 | Be- 7 | 1.28e18 | 10 18888 | Be- 7 | 2.16e18 |
| r_7 At./013 | IRON | } | | 1 | | MS8 | At. x |
| - Land | 78888å | } | | | TEST SI | EC INEM | |
| 2.88614 | Energy Absorber | | | | | | -2.4e-7x |
| 1.58e14 | | ************ | • | Total of A Recoil Ene | | | - 1.8e-7x |
| 1.88e14 | | • | | e e | 1,8162 | | - 1.2e-7% |
| 5.88e13 | | | | 18 | y depth | | 5.9 c -8% |
| <u> </u> | | 866K | 22 | | - The same of the | | |
| Sur Sur | <u>face 58888</u> | 188888 | 15 | 8888 | 288888 | Dep | th (A) |

FIGURE 4-4. CALCULATION OF DEPTH PROFILE OF EACH ENERGY BIN OF BE⁷ RECOILS IN M50

SECTION 5 DATA ACQUISITION SYSTEM

The data which need to be collected during an SLA wear test is simply the number of .amma rays per second emitted by the wearing part and to the initial value of this rate before wear. If one includes the half-life correction then:

$$f = \frac{A}{A_o e^{-\lambda t}}$$

If this measured value of the count rate is then used in conjunction with a previously measured calibration curve, the wear amount can be directly read from the curve.

For example, using the calibration curve of Figure 5-1, if we measure a value of f=92% after 3 days of running, then the wear amount, is 5 microns.

The measurement of this count rate is usually done with the most sensitive gamma ray detector available. In this case a 4 in. or 6 in. diameter Sodium Iodide (NaI) scintillation detector is the best choice.

In a properly designed SLA system, the MaI detector sensitivity can be so high and the source activity so low, that a routine Geiger counter survey will show no activity at all. This is important to appease fears of radiation, however unjustified, by the many persons involved in the operation of a bearing test rig or an aircraft test.

The lowest cost instrumentation is a personal computer which is outfitted with an analog-to-digital converter. This will provide a low cost system for future experiments and can also become the basis for a low cost production unit when the wear monitoring system is deployed in wide use.

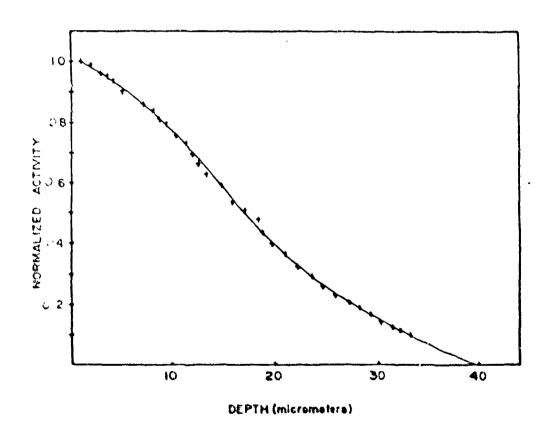


FIGURE 5-1. TYPICAL CALIBRATION CURVE IN SLA WEAR TEST.

The basic system design will include the components in the block diagram shown in Figure 5-2 below. All of the components are commercially available and therefore no engineering risk is involved in assembling such a system. The detector itself will be a 4" x 4" Sodium Iodine detector readily available commercially.

Once the gamma ray spectrum is in the computer, it will be stripped of background and fitted to a gaussian-shaped peak as shown in Figure 5-3. After fitting, the computer can also make the half-life correction, look-up the wear on the stored calibration curve, and display a plot of wear as a function test-time.

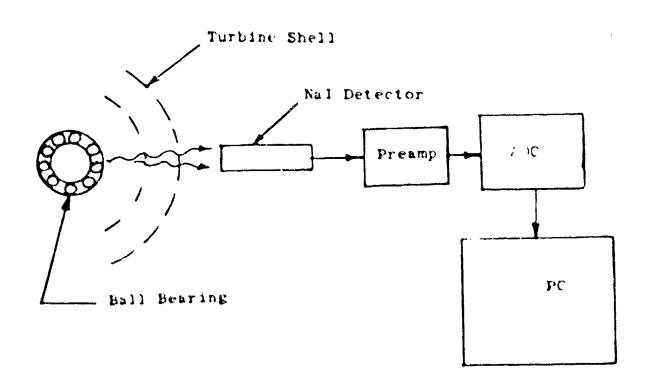


FIGURE 5-2 BLOCK DIAGRAM OF GAMMA RAY DETECTION SYSTEM.

The gamma detection system will be able to performance with both the pin-on-disk units and the bearing test rig with a total initial activity on the parts of less than 0.8 microcurie. This is below the limit of regulation by the NRC. (See Section 8)

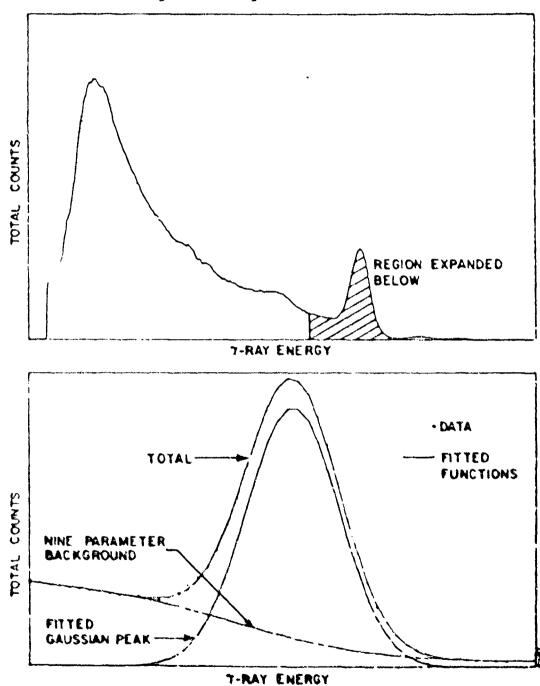


FIGURE 5-3. SPECTRUM OF BE GAMMA RAYS IN NAI DETECTOR (UPPER) AND METHOD OF STRIPPING BACKGROUND

SECTION 6 CALIBRATION TECHNIQUES

In any SLA system, the accuracy of the measurement is limited by the calibration curve. This calibration curve is determined as follows:

- 1. Irradiate another piece of the wear component (the disk or the inside race) with the identical irradiation conditions and geometry as the prime part.
- 2. Artificially remove precise amounts of material. Measure the change in thickness with a precision micrometer and the change in activity content with the nuclear detection system.

This procedure yields a percent activity vs. depth curve such as previously shown in Figure 5-1. In most of the wear measurements of automobile piston rings and cams previously reported, the use of sandpaper was adequate since the activation depth was 30 to 1000 microns and measurement of changes in thickness were several microns (2.5 micron=0.1 mils) for each point on the calibration curve.

In the case of ceramic ball or roller bearings, where the expected wear is only a few microns, sandpaper cannot be used to "wear" away precise amounts on the calibration sample. At Implant Sciences we have developed a method of ion beam milling which can remove precisely known parallel layers of ceramics for calibration purposes.

An experiment was done during this program to measure the milling rate of high energy Xenon ions sputter eroding a Silicon Carbide specimen. We found that 36mA-h of 100keV Xenon directed on a flat surface of SiC milled away precisely 5.0 microns of material. The final measurement of 5.0 microns was done using a thin film profiler with a 0.05 micron accuracy.

Thus, by ion beam milling for 5% of the time (1.8mA-h), we can remove 0.25 microns and sequentially measure the loss of activity. Using this method a calibration curve of count rate observed vs. wear depth (in 1/4-micron increments) can be determined.

The problem of creating an accurate calibration curve is a critical issue in measuring very low levels of wear in bearings. It is believed that no other workers in the world have developed such a precision method using ion milling, and its use in this application could mean the difference between reliable data and uncertain results.

SECTION 7 RADIATION SAFETY CONSIDERATIONS

extremely low and are not subject to NRC regulation, because the levels are below the exempt quantities for the isotopes of interest. Table 7-1 shows that both Be⁷ + Co⁵⁶ needed for this program are below the exempt quantities in NRS regulation, paragraph 30.71. Also the dose rate from these active bearings should be about 0.1 mrem/h on contact and less than 0.02 mrem/h at 6 in. away. When shielded by the pin-on-disk apparatus or the bearing test rig, there should be no measurable radiation using a geiger counter. Ambient background is in the range of 0.01 to 0.02 mrem/h.

TABLE 7-1. EXEMPT QUANTITIES OF RADIOACTIVE MATERIALS

| Schedule B (1) | | Need Radioactivity | |
|---------------------------------------|-------------------|-----------------------|--|
| · · · · · · · · · · · · · · · · · · · | (uCi) | at start of test(pCi) | |
| Be ⁷ | 1 | 0.6 | |
| Co ⁵⁶ | 1 | 0.5 | |
| (1) NRC re | egulation (30.71) | | |
| | | | |

Table 7-2 shows how these levels compare to doses from common sources.

The only NRC regulation mandated during the installation and running of the test program is that a "Caution Radioactive Material" tag must be affixed to the box containing the parts. Also the radiation safety procedures required to assemble the

bearings unto the test rig are minimal or none. The only requirement is that the technician must be made aware that the parts are radioactive, but no special precautions are required.

TABLE 7-2. RADIATION DOSE RATES AND DOSES FROM COMMON SOURCES

| | Dose | |
|---|----------|---------------|
| | (mrem/h) | mrem |
| Potassium-40 naturally occurring in the body (1) | 0.002 | 20/year |
| Natural background dose at sea level(average) (1) | 0.011 | 100/year |
| 3-h jet-plane flight (1) | 0.67 | 2/flight |
| Chest X-ray Source | - | 30/exposur€ |
| Dental X-ray single exposure (2) | - 250 | -450/exposure |

⁽¹⁾ Reference 16

⁽²⁾ Reference 17

^{*0.0118} percent of all potassium is potassium-40

SECTION 8 CONCLUSION AND RECOMMENDATIONS

This program investigated the technical feasibility of using the surface layer activation technique for the measurement of wear rates applicable to all ceramic and hybrid-ceramic ball bearings.

The general technique had to be modified to accommodate the extremely fine wear amounts experienced by ceramic bearings. An important aspect of the work was shown to be adjusting the depth penetration of the implanted isotope (Be⁷) in the thinnest possible layer so that the maximum measurement sensitivity could be obtained. Another important issue that was successfully resolved was the calibration technique that had to be developed to measure wear differences with an accuracy at the 1/4-micron level. Specifically the conclusions reached during this work are:

- 1. The SLA technique, using Be^7 recoil implantation as the nuclear tagging isotope, is a particularly sensitive method of measuring wear in ceramic or metal rolling element bearings in-situ and with 1/4-micron resolution.
- 2. Techniques and software have been developed to calculate the precise isotope implantation concentrations and depths to yield a specific measurement accuracy and wear depth resolution.
- 3. Specific instrumentation and vendors have been identified to purchase and assemble a system for detection of the radiation signal. Also specific accelerator laboratories have been identified to perform the activation service for subsequent programs.
- 4. All of the necessary scientific and engineering issues have been resolved in preparation for implementation of this measurement both in the laboratory and in a bearing test rig of the Air Force's choice.

As a result of these findings, it is recommended that a follow-on program be initiated to move this technology from

the analysis phase into the laboratory. The test program should have the following specific components:

- l. Pin-on-disk measurements of ceramic wear in various load, temperatures and lubrication conditions should be made. These measurements should include various ceramic-on-ceramic and ceramic on bearing steel wear couples.
- 2. The activation techniques necessary to perform should be implemented at an appropriate accelerator laboratory, and the samples should be used to verify the software predictions made in this study.
- 3. A calibration apparatus should be set up and wear calibration curves should be measured for several ceramic materials of interest.
- 4. A specific bearing should be selected for testing in a bearing test rig designated by the Government.
- 5. The wear test data received from the bearing test rig should be correlated to the basic pin-on-disk data generated at the beginning of the program.

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Appendix A Calculation of Expected Count Rates in Detector

The calculation of gamma count rate in a Sodium Iodide detector is straightforward although there are numerous terms in the expression. Since many hundreds of calculations had to be performed in this program to select all of the free parameters, we decided to write a computer program to solve this problem.

In general, the expression for the count rate measured by a detector is

$$A = A_0 \Omega \in e^{-\mu_n \rho_n t_n}$$

where:

A, is the source activity in disintegrations/second

∴ is the solid angle fraction of 4-pi steradians

 ϵ is the detector efficiency of the gamma ray of interest. $e^{-\mu_n}$ of the attenuation coefficient of the nth absorber.

The source in this case is either created by direct activation or by recoil implantation. Recoil implantation of ${\rm Be}^7$ is discussed in Appendix B. Direct activation of Carbon, or Nitrogen in SiC, or ${\rm Si}_3$ N₄, respectively, to form ${\rm Be}^7$ in the surface is calculated using the following expression

$$A_o = \lambda \left(\frac{N_0 P}{M}\right) \frac{t}{8} \times 10^{-27} \int_0^{X_0} \sigma(x) I(x) dx$$

where:

 λ = the radioactive decay constant

 $N_A = Avogadros Number$

f = density of target

M = Atomic weight of target

t = Activation time (in seconds)

Q' = electronic charge

 $C(\lambda)$ = reaction cross section (mb)

I(x) = Beam current (uA)

activation depth = $R(E_0) - R(E_T) = X_0$

The result of two calculations, one for the pin-on-disk case, and one for the bearing test rig case are shown in Tables A-1 and A-2 respectively.

TABLE A-1 CALCULATION OF COUNT RATE IN BEARING TEST RIG (RECOIL ACTIVATION METHOD)

| DETECTOR | ABSORBERS | NUCLEAR REACTION |
|---|---|--|
| Distance = 8 inch Diameter = 6 inch Efficiency = .9 | Steel = .5 inch Si3N4 = .75 inch Ceramic = Ø inch | Sigma = 25 mbarn Depth = 9.999999 micron Beam Current = 10µamp |
| Activity(Microcurie) | Beam Time(Minutes) | Count Rate(cps) |
| 0.031 | 10.53 | 15 |
| Ø. Ø63 | 21.06 | 30 |
| Ø. Ø94 | 31.59 | 45 |
| Ø.126 | 42.12 | 6Ø |
| Ø.157 | 52.65 | 75 |
| 0.189 | 63.18 | 90 |
| 0.220 | 73.70 | 1Ø5 |
| Ø.252 | 64.23 | 120 |
| Ø.283 | 94.76 | 135 |
| Ø.315 | 105.29 | 150 |
| Ø.346 | 115.82 | 165 |
| Ø.377 | 126.35 | 180 |
| 0.409 | 136.88 | 195 |
| 0.440 | 147.41 | 210 |
| 0.472 | 157.94 | 225 |
| 0.503 | 168.47 | 240 |
| Ø.535 | 179.00 | 255 |
| Ø.566 | 189.53 | 270 |
| Ø.598 | 200.06 | 285 |
| 0.629 | 210.59 | 300 |

TABLE A-2 . CALCULATION OF COUNT RATE IN PIN-ON-DISK TEST (DIRECT ACTIVATION METHOD)

| DETECTOR | ABSORBERS | NUCLEAR REACTION |
|---|---|--|
| Distance = 8 inch Diameter = 4 inch Efficiency = .9 | Steel = .062 inch Si3N4 = 0 inch Ceramic = .25 inch | Sigma = 25 mbarn Depth = 9.999999 micron Beam Current = 10µamp |
| Activity(Microcurie) | Beam Time(Minutes) | Count Rate(cps) |
| Ø.Ø26 | 8.54 | 15 |
| 0.051 | 17.07 | 30 |
| 0.077 | 25.61 | 4 5 |
| 0.102 | 34.14 | ۯ |
| 0.128 | 42.68 | 75 |
| Ø.153 | 51.22 | 9Ø |
| Ø.179 | 59.75 | 105 |
| 0.204 | 68.29 | 120 |
| 0.230 | 76.82 | 135 |
| Ø. 255 | 85.36 | 150 |
| Ø.281 | 93.90 | 165 |
| | 102.43 | 180 |
| 0.332 | 110.97 | 195 |
| Ø.357 | 119.50 | 210 |
| 0.383 | 128.04 | 225 |
| 0.408 | 136.58 | 240 |
| 0.434 | 145.11 | 255 |
| Ø.459 | 153.65 | 27Ø |
| Ø.485 | 162.18 | 285 |
| 0.510 | 170.72 | . 30 0 |

Appendix B

Calculation of Recoil Implanted Activity Versus Depth

The calculation of the quantity of radioactive recoil atoms deposited in a catcher sample is extremely complex. It is necessary to follow the reacting particles through each of the interactions and sum the contributions. The problem is made more difficult by the multivalued nature of recoil energy versus angle and the fact that for thick targets, the nuclear reaction can occur for a variety of incoming beam energies. In the following, target refers to the sample struck by incoming beam, and catcher sample refers to the secondary target which receives recoils. The complete set of calculation steps based on the geometry of Figure B-1 is as follows.

- 1. Select initial starting parameters:
 - (a) incoming beam energy
 - (b) angle of incidence
 - (c) reaction of interest
- 2. Iterate on depth-below-surface where reaction takes place.
- 3. Calculate energy of incoming beam at point of reaction.
- 4. Determine angle from point of reaction to point in catcher sample.
- 5. Calculate both energies of recoil atom at this angle and team energy.
- 6. Calculate differential cross section at this angle and beam energy.
- 7. Calculate energy loss through first target before escape to reach catcher sample (both scattered energies).

- 8. Calculate flux of recoils at the two energies incident on the catcher sample.
- 9. Calculate depth distributions of recoil atoms in catcher sample for both energies after correcting for energy loss in pre-absorber foil.
- 10. Repeat items 2-9 for all combinations of reaction location and angle which can occur. Because of cusps in the cross sections at the critical angles, fine steps are required.

This is not a straight forward calculation. Using simplified analytic functions for ion range versus energy, it still requires about 1 1/2 hours of computer time using a 20 MHz, 32-bit personal computer with math coprocessor. Older personal computers would need up to 15 hours to complete the problem.

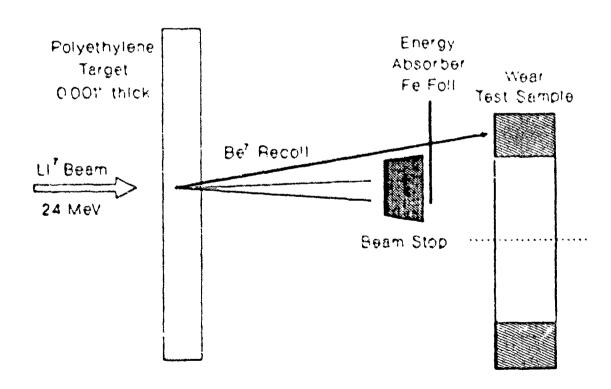


Figure B-1. Geometry of Scattering to Produce Recoil Implantation

Details of the more obscure mathematics involved will now be documented for the interested reader. References are provided for many of the equations used, and no effort is made to show intermediate calculations.

A. Range versus Energy

Computer programs exist to predict the mean range to stop an ion of a given energy in a given target. For light ions, such as protons, deuterons, or alphas, the fastest calculation method is to perform a mathematical fit to data from published tables (6,7). In general it is found that a plot of the log of range versus the log of energy is nearly a straight-line or a (quadratic) over a narrow enough energy band. Thus, one can write

$$log R = A log E + B$$

(3)

R= range E= energy
A,B = constants to be determined

This reduces the problem to a simple case of least squares fitting to tabular data to find A and B. Once obtained, one can use the convenient procedure of determining energy versus thickness traversed by the method of range differences. This is performed as follows:

- 1. Determine range R_{in} at incoming energy E_{in} using equation 1.
- Subtract the thickness of material traversed, dxin

$$R_{out} = R_{in} - dX_{in}$$
 (4)

3. Substitute Rout back into equation 1 and solve for Eout

$$\log E_{\text{out}} = (\log R_{\text{out}} - B)/A \tag{5}$$

This method has the advantage of being fast to calculate although the span of energies accommodated with accuracy is limited. Tabular data for heavy ions has been taken from Implant Science Corporations' Profile Code, which uses a range program by Ziegler (8) with empirical modifications to match ranges calculated by Monte Carlo simulators, such as TRIM. (9)

B. Scattered Flux

The ratio of incoming beam flux versus outgoing recoil flux from a reaction site is given by

$$\frac{flux out}{flux in} = \frac{d\sigma d\theta d\theta \rho dt NAV}{d\Omega 4\pi M_0}$$
 (6)

 $\frac{1}{d_{\perp} 1} = \text{differential cross section in lab coordinates at}$ angle and energy E

 $d\theta$, $d\phi$ = angular spread on catcher sample for solid angle

(| | mass

df = increment of target thickness

NAV = avogadro's number, 6.023x10²³

This equation can be broken into the following components. The differential cross section is the probability of the reaction scattering a recoil into a unit solid angle at some angle. The angle variables give the actual solid angle. The remaining terms are the number of target nuclei that can participate in making a reaction.

C. Differential cross section.

Usually the differential cross section is not published as a function of many energies, although the total cross section is often available. The most difficult part of the overall calculation is this conversion between total and differential cross section. The total cross section of the reaction is shown in Figure B-2.

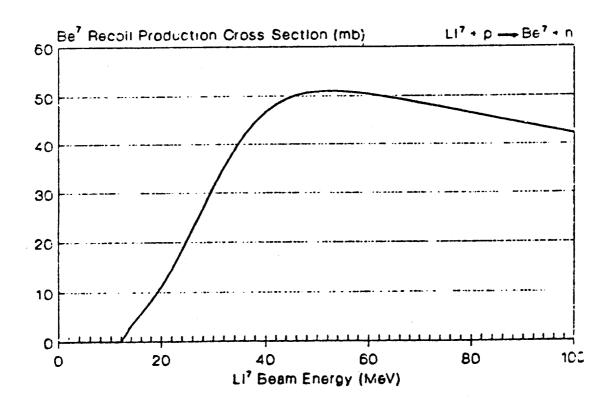


Figure B-2. Cross Section Used for p(Li⁷, Be⁷) n Reaction

Many nuclear reactions are of the type known as compound nuclear reactions. In this model the incoming beam particle merges into the struck nucleus and mixes its energy in completely. Eventually, (after 10^{-20} seconds), recoil product atoms escape. Because of the mixing, information on the direction of the original incoming atom is lost, so the exiting recoil atoms "boil off" in arbitrary directions in the center-of-mass coordinate system. Thus, the differential cross section in the center-of-mass is a constant versus angle. The problem, then, is to convert a constant center-of-mass cross section to laboratory coordinates, integrate the resulting angular distribution, and normalize it to the total cross section as a function of energy. The procedure is a bit tedious.

The necessary equations are listed below. The entire calculation is relativistically correct. A prime indicates center-of-mass variable. (10,11,12)

$$\frac{\left(\frac{d\sigma}{d\Omega}\right)'}{\left(\frac{d\sigma}{d\Omega}\right)_{LAB}} = \frac{b_{s}^{2}c^{2}}{\beta_{s}^{2}c^{2}} / - \frac{\left(W_{B}^{2} - b_{s}^{2}c^{2}\right)\beta^{-2}}{b_{s}^{2}c^{2}\left(1 - \overline{\beta}^{2}\right)} \sin^{2}\theta_{LAB}$$
(7)

One what scattering angle of recoil particle B

$$b_{1}^{2}C^{2} = \frac{\left[Q(a+A+b+B)c^{2}+2N_{KI}Ac^{2}\right]\left[Q(a+A+b+B)c^{2}+2N_{KI}Ac^{2}+4Bbc^{2}\right]^{(8)}}{4\left[2N_{KI}Ac^{2}+(a+A)^{2}c^{4}\right]}$$

e,A,b,B are the masses, a is incoming, A is struck, B is the recoil of interest

$$Q = (a + A + b + B)c^{2}$$
(9)

$$\bar{\beta}^{2} = \frac{W_{KI} \left(W_{KI} + 2ac^{2}\right)}{\left[W_{KI} + (a+A)c^{2}\right]^{2}}$$
(10)

$$W_{B}' = \frac{E^{2} + (Bc^{2})^{2} - (bc^{2})^{2}}{2E'}$$
(11)

$$E' = \sqrt{(ac^2)^2 + (Ac^2)^2 + 2E_1Ac^2}$$
 (12)

$$E_{l}^{2} = (W_{Kl} + ac^{2})^{2} \qquad \qquad \int_{l}^{2} C^{2} = (W_{Kl} + ac^{2})^{2} - \alpha^{2} C^{4} \qquad (13)$$

$$Q_{\mathbf{E}}^{2}e^{2} = E_{\mathbf{B}}^{2} - B^{2}e^{4} \tag{14}$$

$$E_B = (A_1 \pm A_2 A_3)/A_4 \quad \text{both signs physical}$$
 (15)

$$A_1 = [E_1 + Ac^2][E_1Ac^2 + \{(a^2 + A^2 + B^2 - b^2)c^4]$$
 (16)

$$A_2 = \sqrt{\rho^* c^*} \cos \theta_{LAB} \tag{17}$$

$$A_{3} = \sqrt{[E,Ac^{2} + \frac{1}{2}(a^{2} + A^{2} - b^{2} - b^{2})c^{4}]^{-b}B^{2}c^{4} - \rho^{2}c^{2}B^{2}c^{3}sin^{2}\theta_{AB}}$$
 (18)

$$A_{4} = (E_{i} + Ac^{2})^{2} - \rho_{i}^{2} c^{2} \cos^{2}\theta_{LAB}$$
(19)

For normalization we have $C_T(\overline{\mathcal{L}})$ as experimental data and

$$\sigma_{T}(E) = C \int_{0}^{\infty} \frac{d\sigma}{d\Omega_{LAB}} 2\pi \sin \theta_{LAB} d\theta_{LAB}$$
(20)

where \bigcirc_{CRJ} , is determined from

$$\sin \theta_{CRIT} = \sqrt{\frac{Ab}{aB} \left(1 + \frac{Q}{W_{CM}} \right)}$$
 (21)

$$W_{CM} = \frac{AW_{KI}}{(a+A)}$$
 (22)

Equation 7 is summed over as many angles as practical in order to obtain C and get

$$\frac{d\sigma}{d\Omega_{LAB}} = C\frac{d\sigma}{d\Omega_{LAB}} \tag{23}$$

The final differential cross section vs. scattering angle is shown in Figure B-3.

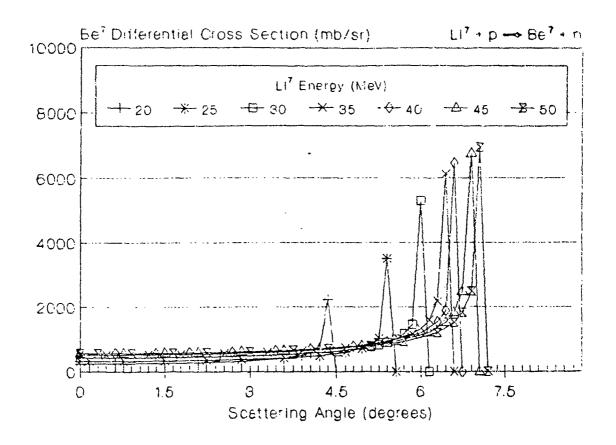


Figure B-3. Differential Cross Section in Lab Coordinates for Several Energies

D. Rinematic Considerations

It has been mentioned that more than one energy is allowed for the recoil scattered atom. This can be seen in Figure B-4, which relates center-of-mass and laboratory particle velocities. This effect occurs whenever $V_{\rm cm}$ $V_{\rm recoil,cm}$. There is also a critical angle, Ocritical' which was given in equation 7, for which only a single $V_{\rm lab}$ recoil can exist. The Lab energy of the recoil at the critical angle is given by (13)

$$E_{CRIT} = \frac{aB}{(a+A)^2} W_{KI} \cos^2 \theta_{CRIT}$$
 (24)

and is not necessarily half-way between the maximum and the minimum values of $W_{\rm B}$. The maximum/minimum lab energies for the recoil atom B are given by the following series of equations where W B comes from equation 5.

$$W_B = \sqrt{\left(\frac{BV_{LAB}}{C}\right)^2 + B^2 c^4} - \beta c^2 \qquad (25)$$

$$W_{KB} = W_B' - \beta c^2 \tag{26}$$

$$B\frac{V_{LAB}}{C} = \frac{B}{C} \left(V_{CM} \pm V_{B}' \right) \tag{27}$$

$$= \frac{AB}{(a+A)a} \sqrt{(W_{KI} + ac^{2})^{2} - a^{2}c^{4}}$$

$$= \frac{AB}{(a+A)a} \sqrt{(W_{KB} + \beta c^{2})^{2} - \beta^{2}c^{4}}$$
(28)

It is important to select the kinematic situation in which $V_{\rm CM}$ $V_{\rm B}$ because then the scattering angle in the lab cannot be greater than the critical angle. This keeps all of the reaction yield constrained to a small band of scattering angles and greatly improves the rate of accumulation of reaction products.

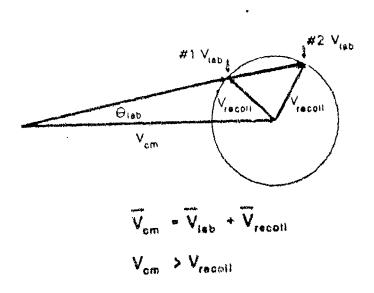


Figure B-4. Graphical Relationship Between Center-of-Mass and Laboratory Velocities of Recoil Atoms. Note that 2 Center-of-Mass Angles and 2 Lab Velocities are Allowed for 1 Lab Angle.